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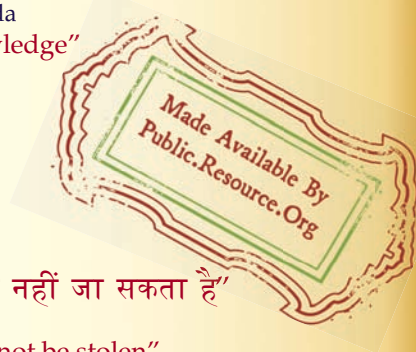
IS 13370 (1992): Linear fatty alcohols for surface active agents [CHD 25: Soaps and other Surface Active Agents]



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भारतीय मानक
सतह सक्रिय अभिकर्मकों के लिए वसीय
एल्कोहल — विशिष्ट

Indian Standard

**LINEAR FATTY ALCOHOLS FOR SURFACE
ACTIVE AGENTS — SPECIFICATION**

UDC 661.72 : 661.185

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 3

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Soaps and Other Surface Agents Sectional Committee had been approved by the Chemical Division Council.

Linear fatty alcohols are used as starting materials for various types of surfactants. The surfactants derived from these alcohols have good emulsifying, dispersing, wetting and detergent properties.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 MARCH 2002
TO
IS 13370 : 1992 LINEAR FATTY ALCOHOLS FOR
SURFACE ACTIVE AGENTS — SPECIFICATION

(*Page 3, clause A-3.1.1*) — Substitute '0.01 *N*' for '0.1 *N*'.

(*Page 3, clause A-3.2*):

a) *Line 3* — Substitute '0.01 *N*' for '0.1 *N*'.

b) *Line 6* — Substitute '0.01 *N*' for '0.1'.

(*Page 4, clause A-4.2, line 1*) — Substitute '20 to 25 g' for '3 to 5 g'.

(CHD 25)

Reprography Unit, BIS, New Delhi, India

Indian Standard

LINEAR FATTY ALCOHOLS FOR SURFACE ACTIVE AGENTS — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for linear fatty alcohols, namely, octanols, decanol, lauryl alcohol, myristyl alcohol, cetyl alcohol, stearyl and mixtures of lauryl-myristyl and cetyl-stearyl alcohol for use in the manufacture of surface active agents.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS	No.	Title
286 : 1978		Methods of sampling and test for soaps (<i>second revision</i>)
1070 : 1992		Reagent grade water (<i>third revision</i>)
2362 : 1973		Determination of water by the Karl Fischer method (<i>first revision</i>)

3 REQUIREMENTS

3.1 Description

The fatty alcohols shall be straight chain even numbered saturated alcohols represented by the general formula $R - OH$, where R is an alkyl group. The carbon chain length of the alkyl group may vary from C_3 to C_{18} .

3.1.1 These may be mixtures of one or more alcohols from the above mentioned range.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A and IS 286 : 1978. Reference to relevant clauses of Annex A and IS 286 : 1978 is given in col 12 and 13 of Table 1.

4 PACKING

4.1 Liquid alcohols shall be packed in closed mouth (MS) drums.

4.2 Flaked alcohols shall be packed in HDPE bags with polyethylene liner.

4.3 Fused alcohols shall be packed in open mouth (MS) drums.

5 MARKING

The containers shall be legibly and indelibly marked with the following information:

- a) Name of the material;
- b) Net mass of the material;
- c) Indication of the source of manufacture;
- d) Batch number or lot number in code or otherwise; and
- e) Month and year of manufacture.

6 SAMPLING

The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Annex B.

7 TESTS

7.1 Tests to evaluate the characteristics prescribed in Table 1 shall be carried out as prescribed in the relevant clauses of Annex A. Reference to relevant clauses of Annex A is given in col 13 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be used in tests.

NOTE – 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Linear Fatty Alcohols for Surface Active Agents
(Clause 3.2)

Sl No.	Characteristic	Types							Cetyl-Stearyl Alcohol	Method of Test, Ref to		
		Octanol	Decanol	Lauryl Alcohol	Lauryl-Myristyl Alcohol	Myristyl Alcohol	Cetyl Alcohol	Stearyl Alcohol		CI No. in	Annex A	
		(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)
i)	Appearance, at 30°C	Liquid	Liquid	Liquid	Liquid	Solid (fused)	Solid (flakes)	Solid (flakes)	Solid (flakes)	II	—	—
ii)	Colour Hazes, <i>Max</i>	10	10	10	10	20	20	20	20	20	—	A-1
iii)	Hydroxyl value	415-423	345-155	295-306	285-295	255-265	225-235	200-210	210-225	205-220	—	A-2
iv)	Acid value, <i>Max</i>	—	0.1	—	—	—	0.3	—	—	—	—	A-3
v)	Saponification value, <i>Max</i>	—	0.5	—	—	—	1.0	—	—	—	—	A-4
vi)	Iodine value, <i>Max</i>	—	0.5	—	—	—	1.0	—	—	—	17	—
vii)	Moisture, percent by mass, <i>Max</i>	—	—	—	—	0.2	—	—	—	—	—	A-5
viii)	Hydrocarbon, percent by mass, <i>Max</i>	—	—	—	1.5	—	—	—	—	—	—	A-6
ix)	Composition by GLC, percent											
	C ₆	0-0.5, <i>Min</i>										A-7
	C ₂	90, <i>Min</i>	0.5	—	—	—	—	—	—	—	—	
	C ₁₀	0-10	90, <i>Min</i>	0-2	0-2	—	—	—	—	—	—	
	C ₁₂	—	0-5	95, <i>Min</i>	70-75	0-5	—	—	—	0-2	—	
	C ₁₄	—	—	0-5	25-30	90, <i>Min</i>	0-5	0-5	0-3	2-7	—	
					0-2	0-5	90, <i>Min</i>	0-5	40-60	25-35	—	
	C ₁₆	—	—	—	—	—	0-5	95, <i>Min</i>	40-60	60-70	—	
	C ₂₀	—	—	—	—	—	—	0-2	0-3	0-2	—	

ANNEX A

(Table 1, Clauses 3.2 and 7.1)

METHODS OF TEST FOR LINEAR FATTY ALCOHOLS FOR SURFACE ACTIVE AGENTS

A-1 DETERMINATION OF COLOUR (HAZEN)

A-1.1 Apparatus

A-1.1.1 APHA (Hazen) Colorimeter

A-1.1.2 Nessler Cylinders

2 cm internal diameter, 12 cm long with marking at 4.7 cm.

A-1.2 Procedure

Fill one Nessler cylinder with water and other with fatty alcohol. Put the colorimeter wheel marked with No. 5, 10, 15, 20, 30, 40, 50, 60 and 70. See through the lens and rotate colorimeter the wheel till both sides of colorimeter show same colour. Note down the reading of colorimeter.

A-2 DETERMINATION OF HYDROXYL VALUE

The hydroxyl value is the number of milligrams of potassium hydroxide equivalent to the hydroxyl content of one gram of sample.

A-2.1 Reagents

A-2.1.1 Acetylating Reagents

Freshly prepared before use by mixing 25 ml of acetic anhydride with 75 ml pyridine.

A-2.1.2 Methanol

A-2.1.3 Isopropanol

A-2.1.4 Alcoholic Potassium Hydroxide — 0.5 N.

A-2.2 Procedure

Weigh 0.5 to 0.6 g of the sample into a stoppered conical flask. Add 5 ml of the freshly prepared acetylating reagent accurately by a pipette. Attach an air condenser and reflux on a steam bath for 1 hour. Add 5 ml of distilled water through the air condenser. Reflux for a further 10 minutes. Cool to room temperature. Rinse the air condenser and the neck of the flask temperature. Rinse the air condenser and the neck of the flask with 25 ml of a neutralized mixture of 2 : 1 methanol : Isopropanol. Titrate against 0.5 N alcoholic potassium hydroxide. Conduct a duplicate and two blanks simultaneously.

A-2.3 Calculation

$$\text{Hydroxyl value} = \frac{(B - S) \times N \times 56.1}{M}$$

where

B = volume in ml of potassium hydroxide solution required for blank,

S = volume in ml of potassium hydroxide solution acetylated sample.

N = normality of potassium hydroxide solution, and

M = mass in g of the sample.

A-3 DETERMINATION OF ACID VALUE

A-3.0 Acid value is the number of milligrams of potassium hydroxide required to neutralize free fatty acids in 1 g of sample.

A-3.1 Reagents

A-3.1.1 Alcoholic Sodium Hydroxide — 0.1 N.

A-3.1.2 Methanol

A-3.1.3 Isopropanol

A-3.2 Procedure

Weigh accurately about 10 g of the sample into a conical flask. Add about 25 ml of a 2 : 1 mixture of methanol : isopropanol previously neutralized with 0.1 N sodium hydroxide to a faint pink phenolphthalein colour. Warm to dissolve the sample and titrate against 0.1 sodium hydroxide to the same faint pink colour.

A-3.3 Calculation

$$\text{Acid value} = \frac{T \times N \times 56.1}{M}$$

where

T = ml of sodium hydroxide required for neutralization,

N = normality of hydroxide solution, and

M = mass in g of the sample.

A-4 DETERMINATION OF SAPONIFICATION VALUE

A-4.0 Saponification value is the number of milligrams of potassium hydroxide required to saponify 1 g of sample.

A-4.1 Reagents

A-4.1.1 Alcoholic Potassium Hydroxide — 0.1 N.

A-4.1.2 Hydrochloric Acid — 0.1 N.

A-4.2 Procedure

Accurately weigh about 3 to 5 g of the sample into a 250-ml stoppered conical flask. Add accurately 40 ml of 0.1 *N* alcoholic potassium hydroxide to the sample from a burette and reflux on a sand bath/mantle with an air condenser for 30 minutes. Titrate against 0.1 *N* hydrochloric acid with phenolphthalein indicator. Conduct a blank estimation simultaneously.

A-4.3 Calculation

$$\text{Saponification value} = \frac{(B - S) \times N \times 56.1}{M}$$

where

B = ml of 0.1 *N* hydrochloric acid required for neutralization of blank;

S = ml of 0.1 *N* hydrochloric acid solution;

N = normality of hydrochloric acid solution, and

M = mass in g of the sample.

A-5 DETERMINATION OF MOISTURE**A-5.1 Outline of the Method**

The method determines the actual water content of the material by titration with Karl Fischer reagent which reacts quantitatively with water.

A-5.2 Procedure

Take about 20 g of material, weigh accurately and determine the moisture content by the procedure given in IS 2362 : 1973.

A-6 DETERMINATION OF HYDROCARBON

The analysis measures the hydrocarbon content in fatty alcohols. The principle in the analysis is the absorption of the fatty alcohols on silicagel, while the hydrocarbons are not absorbed.

A-6.1 Reagents**A-6.1.1 Pentane****A-6.2 Procedure**

Weigh accurately about 2 g of sample into a 100-ml flask and add it 50 ml of pentane to it. If it is an alcohol with a high melting point then it has to be melted first in a steam bath and weighed promptly and, after adding pentane, the mixture has to be heated.

Take a glass tube of 50 cm length and 1.2 cm internal diameter. Fill the bottom of the tube with some glass and press it together with a metal rod. Fill up with powdered silicagel (800-200 mesh) and gently tap the tube against a wooden plate about 30 times until the column is 45 cm full.

Place a 100-ml flask underneath the tube. Transfer the sample solution by a small funnel to a round flask with a long and narrow neck. Put the long necked flask

upside down into the silicagel tube and allow the pentane to drip when the round flask is empty, the sample flask is washed by 50 ml pentane, which is transferred to the long necked flask and dripped through the silicagel tube. This takes about two hours. After this, remove the 100-ml flask in which the pentane, with extracts, is collected and put another 50 ml pentane through the silicagel and collect it in a separate flask.

For C_{18} and other alcohols with a high melting point, the silicagel tube shall be heated with a heating bulb. The heating bulb is placed at a distance from the column sufficient to keep the alcohol in a liquid state. Care should be taken that the pentane does not boil.

After the extraction is complete, evaporate the pentane from the 100-ml flask on the steam bath until about 5 ml solution is left in the flask. Transfer this solution to a dry and weighed (*M*) aluminium bowl. Wash the flask twice with 5 ml of pentane. Keep the aluminium bowl on the steam bath and gently evaporate the pentane. Watch the pentane evaporation closely, and when bubbling ceases, remove the bowl immediately. Keep the bowl in the vacuum desiccator at ambient temperature for 15 minutes. Weigh the aluminium bowl again (*N*). The difference in mass (*N* - *M*) gives the hydrocarbon weight. The second extraction with 50 ml pentane is done in the same way and the hydrocarbon determination should not be more than 1 mg.

A-6.3 Calculation

$$\text{Hydrocarbon, percent by mass} = \frac{\text{mass of hydrocarbon} \times 100}{\frac{\text{mass of sample} (N - M) \times 100}{a}}$$

where

N = mass in g of the residue,

M = mass in g of the aluminium bowl, and

a = mass in g of the sample.

A-7 DETERMINATION OF COMPOSITION BY GAS LIQUID CHROMATOGRAPHY

A-7.1 The percentage of various fatty alcohols in a given sample is determined by gas liquid chromatography. Details of a typical procedure is given below.

A-7.2 Any suitable gas liquid chromatograph may be used.

A-7.3 Details of Operation

Sample injected as fatty alcohol:

Column . 22.86 × 0.317 5 cm/OD SS

Packing : packed with 5 percent SE - 30 on chromosorb WAW DMCS

Sample size : 0.1 to 0.2 microlitres as 50 percent solution in chloroform

Programming : Temperature programming 110°C
(1 Min) – 7°C/Min – 220°C hold

Carrier gas : Nitrogen 40 ml/min gas flow

Detector : Flame ionization detector

Injection Temp : 250°C

Detector Temp : 300°C

NOTE — These conditions may, however, be altered depending upon the availability of the type of the instrument.

ANNEX B

(Clause 6)

B-1 GENERAL REQUIREMENTS FOR SAMPLING

B-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that ullage of about 5 percent is left after pouring in the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and other details, if any.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the containers in each batch shall constitute a separate lot.

B-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected from Lots of Different Sizes

Lot Size <i>N</i> (1)	Sample Size <i>n</i> (2)
Up to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

B-2.3 In order to ensure randomness of selection, the following procedure is recommended for use:

"Arrange all the containers in the lot in a systematic manner and starting from any one count them as 1,2,3, . . . , up to *r* and so on, where *r* is the integral part of N/n (*N* and *n* being the lot size and sample size respectively). Every *r*th package thus counted shall be withdrawn to give a sample for the test."

B-3 COMPOSITE SAMPLE

From each of the containers selected according to **B-2.3**, a representative portion of the material from different parts of the container shall be drawn. From each of these individual portions, an equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample not less than 1 500 g. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample. These shall be transferred to clean containers and labelled with full identification particulars of the sample. Referee samples shall be used in case of any dispute between the purchaser and the supplier.

B-4 TESTS

Tests for the determination of all the characteristics given in **3.1** and Table 1 shall be carried out on the composite sample.

B-5 CRITERIA FOR CONFORMITY

For declaring the conformity of the lot to this specification, the test results on the composite sample shall meet the corresponding requirements specified in the standard.

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